

D R A F T

PUFFING TOBACCO WITH GASEOUS OXIDES

PM #568

Mr. John W. Madures and Mr. Robert Young of Philip Morris Incorporated disclosed to me on May 13, 1970, the results of their investigation into the puffing of tobacco with oxides of nitrogen and sulfur.

The invention is a process for expanding tobacco by impregnating it with nitrous oxide or sulfur dioxide or a source of same and subjecting it to expansion conditions such as heat and/or reduced pressure.

The background of tobacco puffing and the reasons for it have been described in our disclosure PM #562.

It has now been found that nitrous oxide and sulfur dioxide are especially satisfactory puffing agents for tobacco leaf. Nitrous oxide has F.D.A. approval as a general purpose food additive; it is used as a propellant in dairy products (pressurized whipped cream), and for medicinal purposes (anesthetic). It is only moderately flammable, low in toxicity, and leaves no residue. Handling of such material would present almost no safety problems, and the presence of traces in the processed tobacco would be innocuous; at the same time, it is an easy matter to remove substantially all of it from tobacco, and to recover it. It is less expensive than the fluorocarbon types of organic puffing agents and less flammable than some others, for example hydrocarbons and alcohol. Nitrous oxide impregnates tobacco rapidly, much more so than alcohol, ammonia, or ammonium carbonate. For example, under comparable conditions the required exposure times are 2 hrs. for  $N_2O$ , 5 hrs. for  $NH_3$ , and 17 hrs. for ethanol, when the preferred

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impregnation process is used for each. Nitrous oxide has only a slight odor, not objectionable, and thus presents no problem in handling of it or of tobacco containing it such as might be found with certain hydrocarbons, etc. There is no tendency for  $N_2O$  to cause discoloration of tobacco by heat in the presence of oxygen or air, as is the case with ammonia in certain circumstances. Impregnation is a one-stage process, in contrast with ammonium carbonate or bicarbonate (vapor) impregnation which preferably is done in two stages with  $CO_2$  and  $NH_3$ . Moreover, there is not a pronounced heat of absorption evolved with  $N_2O$ , in contrast with the just-mentioned  $NH_3/CO_2$ , or even with  $NH_3$  alone, with the result that treating equipment for  $N_2O$  need not be so sophisticated and expensive.

Sulfur dioxide has many, though not all, of the advantages of nitrous oxide, as well as one the latter does not have, complete nonflammability. It is cheaper than the fluoro-organics and nitrous oxide, can be handled and recovered quite easily, and has approval as a preservative-type food additive. It impregnates tobacco more rapidly than alcohol, ammonia, or ammonium carbonate; under similar circumstances  $SO_2$  requires 2 hrs.,  $NH_3$  5 hrs., and alcohol 17 hrs. Sulfur dioxide is a toxic and highly irritant gas, however, and precautions in handling would be required accordingly. Not more than traces could be tolerated in the tobacco.

For puffing with nitrous oxide, the cured tobacco leaf (usually cut or chopped as for filler) is impregnated by exposure to the gas for a period of 15 to 45 hrs., by spraying with the liquid and exposure for 15 to 45 hrs., or preferably by exposure to the liquified oxide by immersion for 15 to 45 hrs. Preferred exposure times are 15 to 45 hrs (gas), 15 to 45 hrs (spray), and 15 to 45 hrs (immersion). The useful ranges of nitrous oxide concentration in the leaf (dry weight basis) are 1 to 15 %, with the preferred range to 10 %. Moisture content at the time of exposure does not

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seem to be of great importance. Leaf with a normal moisture level of about 12-14% has been used with success.

The puffing processes have been described in detail in our disclosure PM #562. As with other agents, the requirements for puffing are a rapid temperature increase or a rapid pressure reduction around the tobacco, or both. Satisfactory puffing with nitrous oxide-impregnated cut filler has been achieved with a "P.B." Dispersion Dryer (Proctor & Schwarz Co.) operating with air or superheated steam or mixtures of these at inlet temperatures of 400 to 500 °F., and exposure times of 0.50 to 3.0 seconds.

Puffing with sulfur dioxide calls for impregnation by exposure of the leaf, usually cut, to the gas for a period of 10 to 20 minutes, to spray of the liquid and exposure for 10 to 24 hours, or preferably to the liquid SO<sub>2</sub> by immersion for 10 to 24 hours. Preferred exposure times are 10 to 20 minutes (gas), 10 to 24 hours (spray), and 10 to 24 hours (immersion). The useful ranges of sulfur dioxide concentration in the leaf (dry weight basis) are 1 to 10 %, and the preferred range 2 to 5 %. Moisture content at the time of exposure 12 to 14 %.

Satisfactory puffing with sulfur dioxide-impregnated filler has been achieved with a "P.B." Dispersion Dryer operating with air or superheated steam or mixtures of these at inlet temperatures of 400 to 500 °F., and exposure times of 0.50 to 3.0 seconds.

The process of the invention may be illustrated by the following examples.

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EXAMPLE 1

A cured cut cigarette tobacco with about 12% moisture was mixed with a sufficient amount of  $N_2O$  to thoroughly impregnate the tobacco. There was about 20 grams of tobacco to 200 ml. of liquid  $N_2O$  in a Du Voar flask. The  $N_2O$  treated tobacco was equilibrated in a closed container for about two hours and then passed through the P.B. Dispersion Drier at 600°F. with a 45-40 steam to air ratio. At this time the tobacco had expanded and the  $N_2O$  had been completely removed by vaporization.

<u>Tobacco Sample</u>	<u>Density</u>	<u>Code</u>
A. Control E-7	1.100 g/cc	71A
B. $N_2O$ Treated E-7	0.374 g/cc	71B

Filling measurements on the low density treated E-7 produce about a 2:1 filling power ratio expanded to control after equilibration to 12.5 O.V. in tobacco.

EXAMPLE 2

A cured cigarette tobacco in cut filler form was equilibrated at about 12% moisture then submerged in a flask of  $SO_2$  liquid. There was about 25 grams of tobacco and 250 ml of liquid  $SO_2$ . The treated tobacco was equilibrated in this closed container for about two hours and then passed through the P.B. Dispersion Drier at 600°F. with a 45-40 steam to air ratio. At this time the tobacco had expanded and the  $SO_2$  had been completely removed by evaporation.

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<u>Tobacco Sample</u>	<u>Density</u>	<u>Code</u>
A. Control E-7 Bright	1.14 gm/cc	1-A
B. SO <sub>2</sub> Treated E-7 Bright	0.38 g/cc	1-B

Filling measurements on the low density treated E-7 tobacco produce about a 2:1 filling power ratio expanded to control after equilibration to 12.5 O.V.

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